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(54) **Epoxy powder coating compositions containing calcium oxide.**

(57) Epoxy powder coating compositions suitable as pipe coatings and which exhibit reduced foaming upon heat curing are obtained by including calcium oxide in the compositions.

EPOXY POWDER COATING COMPOSITIONS  
CONTAINING CALCIUM OXIDE

This invention relates to epoxy powder coating compositions containing calcium oxide.

Epoxy powder coating compositions are well known and widely used. Examples of such compositions are described in U.S. Patents 4,122,060 and 4,322,456.

Calcium oxide has been used in various rubber and plastics compositions particularly to eliminate the porosity that develops during vulcanization of rubber, for example. Hitherto, it has not been proposed to include calcium oxide in epoxy powder coating compositions.

According to the present invention, there are provided heat-curable epoxy powder coating compositions comprising epoxy resin and filler including calcium oxide in an amount sufficient to reduce foaming when the composition is heat cured. Substrates may be coated with such compositions by applying the composition to the substrate and fusing and curing the composition to a film.

The presence of calcium oxide in the coating assists in reducing foaming which occurs on heat-cure of known compositions not containing calcium oxide.

Preferably, the calcium oxide is treated with a hydrophobic material to reduce hydrolysis. Typical hydrophobic agents include wax and oil. Treated calcium oxide suitable for use in this invention is commercially available. The amount of calcium oxide that is included in the composition is the amount that is sufficient to reduce foaming of the particular epoxy composition. Generally, that amount is from 1 to 10% by weight of the total composition, preferably from 2 to 8% by weight; in certain applications, as little as 3% by weight has been found to be totally satisfactory.

An essential component of the compositions of the invention is a solid epoxy resin. In general, any solid epoxy resin is suitable. Particularly preferred are epoxy resins which are the condensation products of bisphenol-A and epichlorohydrin. Also suitable are epoxy novolac resins. One or more such epoxy resins can be used as well as epoxy resins of different chemical constitution.

Preferably, the compositions of the invention include resinous curing agents which are reactive with epoxy resins. Suitable materials of this type include novolac resins and, in particular, polyhydric phenols; suitable polyhydric phenols are described in U.S. Patent 4,322,456. Preferred polyhydric phenols include those materials described in U.S. Patent 4,322,456 prepared by reacting an excess of bisphenol with a diglycidyl ether of bisphenol in the presence of a suitable catalyst, and resins obtained by reacting a bisphenol with an epoxy novolac resin.

The compositions generally include catalysts in amounts from 0.001 to 10% and preferably from 0.05 to 5% by weight, based on the weight of the epoxy-containing compound and the curing agent.

Effective catalysts include phosphonium compounds and imidazoles as described in U.S. Patent 4,322,456, but any suitable catalyst can be used.

Pigments, fillers, dyes, flow control agents and other additives can be included in the compositions of the invention. Typical fillers (other than calcium oxide) are barium sulfate, calcium silicate, calcium meta silicate, and calcium carbonate. Calcium oxide can be used as the only filler or can be used in conjunction with other fillers.

The components of the composition are advantageously combined by melt blending, extruding the melt and pulverizing to achieve a powder with a particle size predominantly less than 150 microns. Preferably, more than 90% and most preferably more than 96% of the particles are smaller than 150 microns.

Coatings are prepared from the compositions of the invention by applying the composition to a substrate and heating to effect curing. The substrate may be heated either before or after application of the powder coating composition. Typically in pipe coating applications for which the compositions of the invention are particularly suitable, the pipe is heated and the powder is applied to the heated pipe. Curing is conducted at temperatures sufficiently high to give reasonable cure times; generally these temperatures are from 130 to 300°C. The thickness of the applied coating depends largely on the ultimate use of the coated article; thicknesses are generally from 200 to 1000 microns and preferably from 300 to 500 microns.

The invention is illustrated by the following Examples, in which all parts are by weight.

#### EXAMPLE 1

This example illustrates a representative composition in accordance with the present invention.

The following materials, except the silica, are melt blended and extruded at a temperature of 90 to 100°C, pulverized into a powder (greater than 96% below 150 microns) and the silica is dry blended with the powder.

	CONTROL	EXAMPLE 1
DER 664U, Epoxy Resin	310.00	310.00
DER 672U, Epoxy Resin	320.00	320.00
XD8062.01 Curing Agent	138.00	138.00
EPON P104 Curing Agent	5.00	5.00
BYK Powder Flow VP360P, Flow Additive	2.00	2.00
Pigments	21.20	21.20
Mica	23.00	23.00
Calcium Meta Silicate	180.40	100.40
CALOXOL CP2		80.00
AEROSIL R972 Silica	0.40	0.40
	1000.00	1000.00

DER 664 U is a bisphenol-A epoxy resin having an epoxide equivalent weight of 875-975 available from Dow.

DER 672 U is a novolac-modified bisphenol-A epoxy resin having an epoxide equivalent weight of 750-850 available from Dow.

XD 8062.01 is a resinous phenolic curing agent containing 2-methyl imidazole available from Dow.

Epon P104 is a resinous epoxy curing agent of the accelerated dicyandiamide type available from Shell.

Byk Powder Flow VP 360P is a levelling agent for coatings comprising an acrylic polymer absorbed on silica available from Byk-Mallinckrodt.

Caloxol CP2 is calcium oxide coated with mineral oil available from John and E. Sturge Ltd.

Aerosil R972 is silica having an average particle size of 20 millimicrons available from Degussa.

#### EXAMPLE 2

The composition of Example 1 containing calcium oxide was compared to the Control (without calcium oxide) by coating the compositions onto metal and evaluating the coatings.

Hot rolled steel was grit blasted, degreased and cleaned with acetone. The samples were preheated for 15-20 minutes at 274°C resulting in a 246°C metal temperature. The powders were coated to a film build of 300-400 microns and postcured for 5 minutes at 274°C.

The samples were evaluated for voids, impact, bend, hot water soak and cathodic disbondment and rated on a scale of 1 to 10 in which 10 is the best rating. Results are summarized in the Table below.

Void ratings were made by visual microscopic examination. Impact and bend tests were in accordance with ASTM methods. The hot water soak test was conducted by soaking the samples in water at 85°C for 4 weeks and evaluating the samples weekly for changes in

film and adhesion. Cathodic disbondment (cell) was according to British Gas Specification CW6. Cathodic disbondment (tank) is an internal laboratory variation on the British Gas cell method in which multiple samples are tested at one time in a tank.

TABLE

	<u>VOID</u>	<u>Impact</u>		<u>Bend</u>		<u>Hbt Water Soak</u>	<u>Cathodic Disbondment</u>		<u>TOTALS</u>
		<u>R.T.</u>	<u>-40°C</u>	<u>R.T.</u>	<u>-40°C</u>		<u>TANK</u>	<u>CELL</u>	
CONTROL	4.5	7.5	3	6.6	4.8	5.9	7.9	8.6	48.6
EXAMPLE 1	9	10	5.5	9.6	6.6	6.4	6.5	7.6	61.2

It is evident that the sample coated with the composition of Example 1 containing calcium oxide exhibited significantly better void and hot water soak ratings as well as an improved overall total rating.



## CLAIMS:

1. A heat-curable epoxy powder coating composition comprising an epoxy resin and a filler including calcium oxide in an amount sufficient to reduce foaming when the composition is heat cured.
2. A composition according to claim 1, containing from 1 to 10% by weight of calcium oxide, based on the total composition.
3. A composition according to claim 2, containing from 2 to 8% by weight of calcium oxide, based on the total composition.
4. A composition according to any one of claims 1 to 3, in which the calcium oxide is coated with a material which retards absorption of water.
5. A composition according to claim 4, in which the calcium oxide is coated with oil or wax.
6. A composition according to any one of claims 1 to 5, which includes a polyhydroxy curing agent.
7. A composition according to any one of claims 1 to 6, in which the epoxy resin is a bisphenol A-epichlorohydrin epoxy resin.
8. A method for coating a substrate comprising the steps of applying to the substrate a composition according to any one of claims 1 to 7, and fusing and curing the composition to a film on the substrate.

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# EUROPEAN SEARCH REPORT

0104719

Application number

EP 83 30 3961

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. *)
X	US-A-3 039 987 (IRVING N. ELBLING) * claims; column 4, lines 28-68 *	1,2,3 7	C 08 L 63/00 C 09 D 3/58
X	DE-A-2 254 437 (ASAHI KASEI KOGYO) * claims 1,2,3; example 2 *	1	
X	CHEMICAL ABSTRACTS, vol. 80, no. 20, May 20, 1974, page 39, abstract no. 109394j, COLUMBUS, Ohio (US) & JP - A - 73 84 198 (HITACHI LTD) (November 8, 1973) * the whole abstract *	1	
Y	CHEMICAL ABSTRACTS, vol. 96, 1982, page 93, abstract no. 8254p, COLUMBUS, Ohio (US) & SU - A - 856 813 (F.A. GORSHKOV et al.) (August 31, 1981) * the whole abstract *	1	TECHNICAL FIELDS SEARCHED (Int. Cl. *)  C 09 D C 08 L
A	US-A-3 442 849 (IRVING TASHLICK et al.) * claims *	1	
A	US-A-3 582 517 (JOHN G. BAILEY et al.) * claims *	1	
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 02-12-1983	Examiner MALHERBE Y.J.M.
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	

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DOCUMENTS CONSIDERED TO BE RELEVANT			Page 2
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 3)
L	CHEMICAL ABSTRACTS, vol. 88, 1978, page 106, abstract no. 172008s, COLUMBUS, Ohio (US) & JP - A - 77 137 437 (MITSUI PETROCHEMICAL INDUSTRIES, LTD.) (November 16, 1977) * the whole abstract *  -----	1	
			TECHNICAL FIELDS SEARCHED (Int. Cl. 3)
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 02-12-1983	Examiner MALHERBE Y.J.M.
<b>CATEGORY OF CITED DOCUMENTS</b>			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons  & : member of the same patent family, corresponding document	

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